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“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 3200 (2001): Chemical Analysis of Cryolite [MTD 13: Ores and Raw Materials]



“ज्ञान से एक नये भारत का निर्माण”

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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
क्रायोलाइट का रासायनिक विश्लेषण
(दूसरा पुनरीक्षण)

Indian Standard
CHEMICAL ANALYSIS OF CRYOLITE
(*Second Revision*)

ICS 71.040.40; 73.060.40

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 3200 was first published in 1965 covering the determination of fluorine, sodium, total aluminium, Sulphates, phosphorus pentoxide, alumina and loss on ignition in synthetic and natural cryolites. However, earlier while taking up for the first revision, committee decided to split the standard in 10 parts. Accordingly, Parts 2, 3 and 8 of the standard have already been published and Parts 1, 4, 5, 6, 7, 9 and 10 were under different stages.

Later on, it had been decided by the Bureau of Indian Standards as a policy decision that all the parts will be merged into one comprehensive standard. Therefore, all the parts of IS 3200 have been merged into this standard which on publication would supersede the earlier parts already published .

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CHEMICAL ANALYSIS OF CRYOLITE

(Second Revision)

1 SCOPE

This standard describes the method for chemical analysis of cryolite.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
3191 : 1968	Methods of sampling of cryolite and aluminium trifluoride

3 SAMPLING

The sample shall be drawn and prepared in accordance with the method prescribed in IS 3191.

4 QUALITY OF REAGENTS

Unless specified otherwise, reagent grade water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF FLUORINE BY DISTILLATION METHOD

5.1 Outline of the Method

Fluorine is distilled off by treatment of the sample with sulphuric acid and titrated with standard thorium nitrate solution.

5.2 Apparatus

The distillation apparatus (Fig. 1) consists of a 250 ml distillation flask (*A*), side arm of which is connected to a condenser (*B*). The neck of the flask is fitted with a two-holed rubber stopper carrying a thermometer (*C*) and 4 mm glass tube (*D*) connecting the flask to a steam generator (*E*). The latter is equipped with two discharges, one tube (*F*) to release steam and the other tube (*D*) to deliver steam into the distillation flask (*A*) and a safety tube about 90 cm. The distillate is collected in a conical flask (*G*)

5.3 Reagents

5.3.1 Sodium Carbonate, Solid.

5.3.2 Dilute Sulphuric Acid, 1:1 (v/v).

5.3.3 Alizarin Red S, 0.1 Percent (m/v).

5.3.4 Methyl Blue, 0.05 Percent (m/v).

5.3.5 Sodium Hydroxide Solution, 2 Percent (m/v), 1N.

5.3.6 Dilute Hydrochloric Acid, 1:50 (v/v).

5.3.7 Thorium Nitrate Solution (0.025 M)

Dissolve 13.806 g of thorium nitrate [$\text{Th}(\text{NO}_3)_4 \cdot 4\text{H}_2\text{O}$] in water and make up to one litre. Mix well, Standardize against standard sodium fluoride solution.

5.3.8 Standard Sodium Fluoride Solution (1 ml = 1 mg of F)

Take 2.211 g of sodium fluoride and fuse with 0.2 g of sodium carbonate. Transfer the contents to the distillation flask and distill off the solution. Transfer the distillate to one litre volumetric flask and store in a polyethylene bottle.

5.4 Procedure

5.4.1 Take 0.2 000 g of sample in a platinum crucible, and 2 g of sodium carbonate and mix carefully with the platinum wire. Place the crucible in an electric furnace and fuse the material at $800 \pm 20^\circ\text{C}$. Keep at this temperature for 20 minutes. Cool and transfer the fused mass to distillation flask (*A*) and add 60 ml of dilute sulphuric acid (1:1).

5.4.2 Place a conical flask (*G*) under the condenser. Connect the apparatus as shown in Fig. 1. Distill the solution until the boiling point reaches about 140°C . While this is being done; heat the water in the steam generator (*E*) to boiling but do not allow the steam to get in the flask (*A*). When the temperature reaches 165°C admit steam into the flask (*A*). Maintain the temperature at about 140°C by adjusting the steam flow and heating of the flask (*A*). Distill at such a rate that no fumes or mist escape from the conical flask. Continue distillation until nearly 450 ml are collected in a conical flask (*G*). Transfer to a 500-ml volumetric flask.

5.4.3 Cool and make the volume up to the mark and mix well. Pipette out 25 ml aliquot into a 250-ml

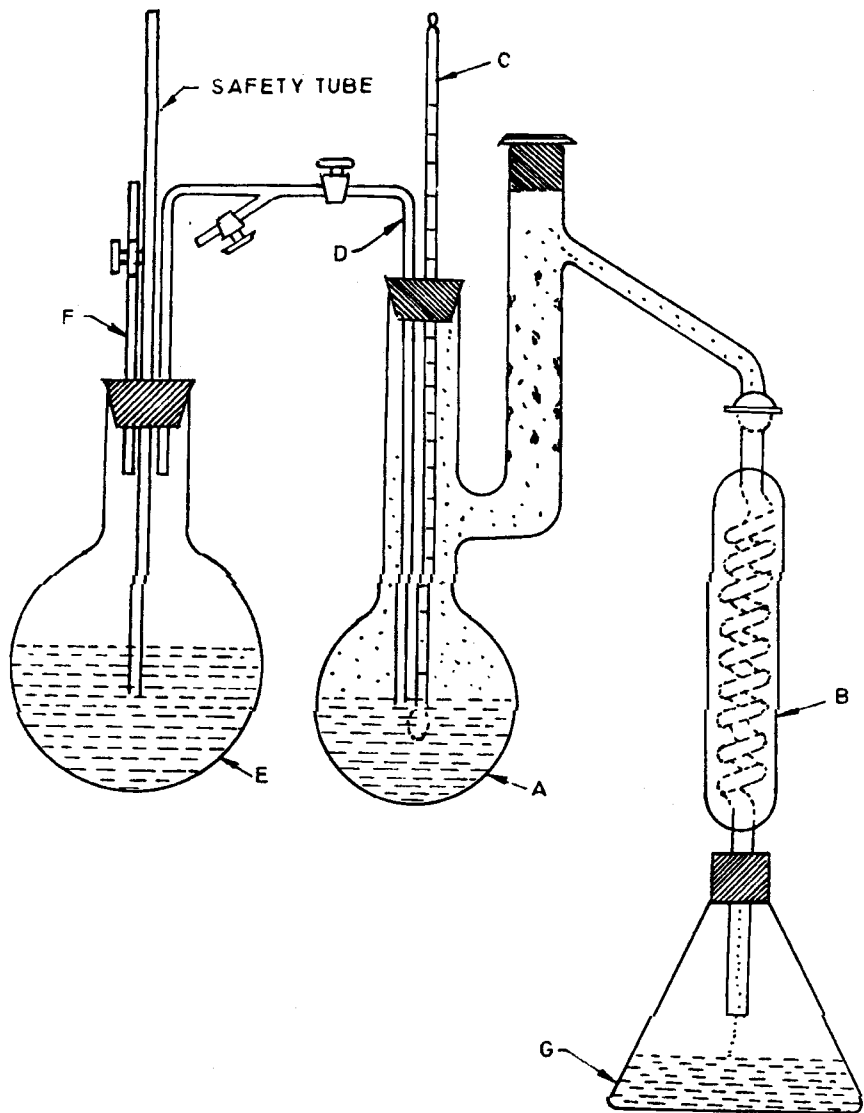


FIG. 1 APPARATUS FOR DISTILLATION OF FLUORINE

beaker. Add 50 ml water, 10 drops of alizarin red S and mix. Add sodium hydroxide solution, slowly with constant stirring until the solution becomes pink (pH will be from 6.6 to 6.8). Add dilute hydrochloric acid drop by drop with constant stirring until the solution becomes yellow (pH of solution will be between 3.5 to 4.0). Add 10 drops of Methylene blue indicator to the solution. Add 2 ml of buffer solution and titrate with standard thorium nitrate solution with constant stirring until a permanent blue violet end point is reached. The colour intensity at the end point should be kept in mind and the titration should be stopped exactly at the same colour intensity for both the thorium nitrate standardization and the test solution.

5.5 Calculation

5.5.1 Fluorine (F) = $\frac{A \times B}{C} \times 100$ percent by mass

where

- A = volume in ml, of standard thorium nitrate solution;
- B = fluorine equivalent of the standard thorium nitrate solution in g/ml; and
- C = mass in g, of the sample represented by the aliquot taken.

5.5.2 Cryolite, percent = $F \times 1.842$

6 DETERMINATION OF SODIUM BY ATOMIC ABSORPTION SPECTROMETRIC (AAS) METHOD

6.1 Outline of the Method

The sample is dissolved in hydrochloric acid and perchloric acid and sodium is determined by atomic absorption spectrometer at 589 nm.

6.2 Reagents

6.2.1 Hydrochloric Acid, $rd = 1.16$ (Conforming to IS 2655X).

6.2.2 Perchloric Acid, 70 Percent (m/v).

6.2.3 Nitric Acid, $rd = 1.42$ (Conforming to IS 264).

6.2.4 Potassium Bromide Solution, 3 Percent (m/v).

6.2.5 Standard Sodium Solution (1 000 µg/ml)

Dissolve 2.542 g of sodium chloride in 200 ml of double distilled water. Dilute to 1 litre in a volumetric flask.

6.3 Apparatus

6.3.1 Atomic Absorption Spectrometer

Equipped with a monochromatic radiation source, such as sodium hollow cathode lamp, a monochromatic to isolate the 589 nm resonance line, an atomization source such as a burner and a read out device.

6.3.2 Operating Parameters

6.3.2.1 Sodium hollow cathode lamp

6.3.2.2 Wavelength, 589 nm

6.3.2.3 Flame, air — acetylene

6.3.2.4 Bandpass — as specified by the manufacturer.

NOTE — Other operating parameters to be followed according to manufacturers instructions.

6.4 Procedure

6.4.1 Test Portion

Weigh to the nearest 0.001 g, 0.1 g of the sample. Transfer it to a platinum crucible.

6.4.2 Dissolution of the Test Portion

6.4.2.1 Add 10 ml of hydrochloric acid, 5 ml of perchloric acid and one or two drops of nitric acid and fume strongly till dissolution is complete. Add 10 ml of hydrochloric acid and digest again. Cool, filter and transfer into 100 ml volumetric flask. Wash the residue thoroughly and transfer the washings into above volumetric flask and make up the volume.

NOTE — While filtering the solution, the filter paper should be made free from sodium by washing it with dilute nitric acid and

then with distilled water in order to avoid any possibility of sodium ions in the solution through filter paper.

6.4.2.2 Transfer 5 ml aliquot (6.4.2.1) into 250-ml volumetric flask. Add 15 ml of hydrochloric acid and 25 ml of potassium bromide solution and make up to the volume.

6.4.3 Preparation of Calibration and Blank Solutions

6.4.3.1 Calibration Solutions

Prepare 0, 5, 10, 15, 20 and 50 µg/ml standard solution by taking corresponding aliquots into one litre volumetric flask. Add 75 ml of hydrochloric acid and 100 ml of potassium bromide solution. Transfer immediately these solutions into polyethylene bottles.

6.4.3.2 Prepare a blank solution using the same quantity of all reagents and following the procedure as specified in 6.4.2.

6.4.4 Adjustment of the Atomic Absorption Spectrometer

Follow the instructions of the manufacturer in preparing the instrument. Switch on the instrument and the sodium hollow cathode lamp. Fit the correct burner for air-acetylene flame and light the appropriate flame (fuel-lean). Wait for about 20 minutes for stabilization. Set the wavelength at 589 nm. Optimize instrument response by adjusting the wavelength, fuel, air, burner and nebulizer while aspirating the highest calibration solution. As the sensitivity varies from instrument to instrument the concentration of the standard series and of the test solution should be adjusted accordingly. At the same time check the linearity of the calibration curve.

Aspirate water and one of the calibration solution repeatedly to ensure that there is no drift of absorbance. Finally aspirate water and set the absorbance to zero reading.

6.4.5 Atomic Absorption Measurement

6.4.5.1 Aspirate first the blank solution and then the calibration solution in increasing order, aspirating water between each aspiration of the solution and record the absorbance reading. Then aspirate the test sample and note the absorbance. Each aspiration should be made at least three times and take the average value. Solids which build up on the burner slit must be removed, otherwise they will cause a decrease of sensitivity.

6.4.5.2 Prepare a calibration curve by plotting the obtained absorbance values of the calibration solutions corrected for the blank against the concentration, in µg/ml. Obtain the concentration of test sample in µg/ml from the calibration curve.

6.4.6 Calculation

$$\text{Sodium, percent by mass} = \frac{A}{B} \times \frac{1}{100}$$

where

- A = concentration in $\mu\text{g/ml}$, of sodium; and
 B = mass in g, of the sample represented by the aliquot taken in the final test solution.

7 DETERMINATION OF SODIUM BY FLAME PHOTOMETRIC METHOD

7.1 Outline of the Method

The sample is dissolved in hydrochloric acid and sodium is determined by flame photometer.

7.2 Apparatus

Flame photometer fitted with atomizer-burner constructed so as to excite emission at the 589 nm sodium line.

7.3 Reagents — Same as given in 6.2.

7.4 Procedure

7.4.1 Take 5 ml aliquot from the main solution (6.4.2) into 250-ml volumetric flask. Add 15 ml of hydrochloric acid and 25 ml of potassium bromide solution and make up the volume.

7.4.2 Blank

Prepare a reagent blank using 15 ml of hydrochloric acid and 25 ml of potassium bromide diluting into 250 ml in a volumetric flask.

7.4.3 Photometric Measurements

Switch on the photometer in advance and allow to stabilize. Adjust the sensitivity of the apparatus and opening of the slit according to the characteristics of the apparatus to ensure a band pass of not more than 6 nm, centered on emission maximum.

7.4.4 Calibration Curve

Atomize the standard calibration solutions (6.4.3.1) and prepare the calibration curve for μg of sodium per millilitre against absorbance values (after deduction of blank value). Convert the absorbance value of sample into $\mu\text{g/ml}$ with the calibration curve and calculate the percentage of sodium in cryolite as indicated under 6.4.6.

8 DETERMINATION OF TOTAL ALUMINIUM BY COMPLEXOMETRIC METHOD

8.1 Outline of the Method

The sample is fused with potassium pyrosulphate and

the fused mass is extracted with water and concentrated hydrochloric acid. Aluminium in the above solution is determined complexometrically.

8.2 Reagents

8.2.1 Potassium Pyrosulphate — Solid.

8.2.2 Hydrochloric Acid, rd = 1.16 (Conforming to IS 265).

8.2.3 Ammonia Solution — Specific Gravity 0.9.

8.2.4 EDTA (0.05 M)

Dissolve 18.64 g of the disodium salt of EDTA in water and transfer to a 1 000 ml volumetric flask, dilute to the mark and mix.

8.2.5 Methyl Red Indicator Solution, 0.1 percent (m/v) in rectified spirit.

8.2.6 Buffer Solution (pH 5.3)

Dissolve 21.5 g of sodium acetate in 300 ml water, add 2 ml glacial acetic acid and dilute to one litre.

8.2.7 Dilute Hydrochloric Acid, 1:1 (v/v).

8.2.8 Standard Zinc Solution (0.05 M)

Dissolve 3.26 g of electrolytic zinc granules 99.95 percent purity in dilute hydrochloric acid (1:1). Evaporate the solution to about 1.5 ml and dilute with water. Add 50 ml buffer solution, mix well and make up the volume to one litre. Standardize against standard aluminium solution.

8.2.9 Standard Aluminium Solution (0.05 M)

Weigh accurately 1.349 0 g of pure aluminium wire in a 500 ml beaker containing 50 ml of water and 50 ml hydrochloric acid. Heat the solution to dissolve the metal. Cool and dilute the solution to 1 000 ml in a volumetric flask. This solution contains 1.35 mg of aluminium per ml.

8.2.10 Xylenol orange solution (0.5 percent) (m/v) Prepared in water acidified with 2 drops of dilute hydrochloric acid (40 percent).

8.2.11 Ammonium Fluoride — Solid.

8.3 Procedure

8.3.1 Fuse 0.5 g of the sample with 5 g of potassium pyrosulphate in a platinum dish until abundant white fumes are given off. Dissolve the fused mass in 150 ml of water and 10 ml of hydrochloric acid and make up to 500 ml.

8.3.2 Pipette out 50 ml of aliquot (8.3.1) to a 250 ml beaker and add 25 ml of 0.05 M EDTA solution and a drop of methyl red indicator. Neutralize the solution with dilute ammonia solution until the colour of the

solution just turns yellow. Add 15 ml buffer solution and boil for about 2 minutes. Cool to room temperature and titrate with standard zinc solution (8.2.8) using 2 to 3 drops of xylenol orange indicator. The colour changes from yellow to red. Add about 2 g of ammonium fluoride to the solution and boil for about 2 minutes. Add 10 ml buffer solution. Cool to room temperature and then titrate with standard zinc solution adding a drop of xylenol orange indicator. The end point is from yellow to pinkish red. The percentage of aluminium is calculated from the titre value of second titration with zinc solution.

8.4 Calculation

$$\text{Total Aluminium, percent by mass} = \frac{A \times B}{C} \times 100$$

where

- A = Volume in ml, of zinc solution required for second titration;
- B = Aluminium equivalent in g, per ml of zinc solution; and
- C = Mass in g, of the sample as represented by the aliquot taken.

9 DETERMINATION OF MOISTURE BY GRAVIMETRIC METHOD

9.1 Outline of the Method

A known weight of powdered sample is dried at 110°C to constant weight and moisture content is calculated from the loss in weight of the sample on drying.

9.2 Procedure

9.2.1 Weigh accurately 25 g of the sample and transfer to petri dish.

9.2.2 Place the petri dish in the oven, raise the temperature to $105 \pm 5^\circ\text{C}$ and dry to constant mass.

9.2.3 Calculation

$$\text{Moisture, percent by mass} = \frac{m_2 - m}{m_2 - m_1} \times 100$$

where

- m_1 = mass of petri dish,
- m_2 = mass of petri dish + sample before drying, and
- m = mass of petri dish + sample after drying.

NOTE — All determination carried out on sample as received should be corrected for moisture content.

10 DETERMINATION OF LOSS OF IGNITION

10.1 Outline of the Method

Finely powdered and dried sample is calcined in the

furnace at a temperature of 550°C to constant mass. Loss in mass of sample is calculated.

10.2 Reagents

10.2.1 Magnesium Perchlorate — Solid.

10.2.2 Caustic Soda, Pellets

NOTES

- 1 The above reagents are recommended for use as desiccants.
- 2 Caustic Soda is to be changed from time to time.

10.3 Procedure

10.3.1 Weigh 2 500 g of the sample into a tared platinum crucible with lid.

10.3.2 Place the crucible with lid in the furnace and raise the temperature slowly to 550°C and heat to constant mass.

10.3.3 Remove the platinum crucible from the furnace, cool in a desiccator and weigh. Repeat the process till constant mass is obtained.

10.3.4 Calculation

$$\text{Loss on ignition (at } 550^\circ\text{C), percent by mass} = \frac{m_1 - m_2}{2.5} \times 100$$

where

- m_1 = mass in g, of empty crucible + 2.5 g sample; and
- m_2 = mass in g, of crucible + sample after heating.

11 DETERMINATION OF IRON BY O-PHENANTHROLINE (PHOTOMETRIC METHOD)

11.1 Outline of the Method

The sample is decomposed by fusion with soda-boric acid. The iron in solution is reduced to the ferrous state with hydroxylamine hydrochloride. After adjustment of pH, O-phenanthroline is added and the photometric measurement of orange-red coloured complex is made at 490 nm.

11.2 Reagents

11.2.1 Boric Acid, Solid.

11.2.2 Sodium Carbonate, Solid.

11.2.3 Dilute Sulphuric Acid, 1 : 1 (v/v).

11.2.4 Tartaric Acid, 10 Percent (m/v).

11.2.5 2:4 Di-nitrophenol Indicator, 0.1 Percent (m/v).

11.2.6 Ammonium Hydroxide Solution

Mix together 120 ml of water and 80 ml of ammonium hydroxide (rd = 0.90).

11.2.7 Dilute Hydrochloric Acid, 1:1 (v/v).

11.2.8 Hydroxylamine Hydrochloride, 1 Percent (m/v).

11.2.9 O-Phenanthroline Solution

Dissolve 0.2 g of ortho-phenanthroline monohydrate in 90 ml of water with stirring and gentle heating. Cool and dilute to 100 ml with water.

11.2.10 Standard Iron Solution (1 ml = 0.1 mg of Fe)

Dissolve 0.100 g of electrolytically pure iron in dilute hydrochloric acid (1:1) and make up to 1 litre.

11.3 Procedure

11.3.1 Fuse one gram of sample with 2 g of boric acid and 4 g of sodium carbonate in a platinum crucible. Transfer the solidified melt to a 250 ml beaker. Remove all traces of melt from the crucible with hot water and add it to the beaker. Dilute to about 75 ml with water, add 10 ml of dilute sulphuric acid (1:1) and heat to dissolve the salts. Transfer to a 250 ml volumetric flask, cool, dilute to the mark with water and mix well.

11.3.2 Pipette 50 ml of aliquot (11.3.1) into a 100 ml beaker. Place in a water bath at 55 to 60°C. Stir and allow the beaker to remain for about 5 minutes.

11.3.3 Add 10 ml of tartaric acid and 0.5 ml of 2:4 di-nitrophenol indicator to the solution. Add ammonium hydroxide solution dropwise with constant stirring until the solution assumes a slight yellow colour. Add dilute hydrochloric acid (1:1) dropwise and with constant stirring until the yellow colour is just discharged. The pH at this stage should be between 3.5 to 4.0.

11.3.4 Add 5 ml of hydroxylamine hydrochloride solution and 5 ml of O-phenanthroline solution. Stir well and cool to room temperature. Transfer to a 100 ml measuring flask and make up to volume with water. Mix well and allow to stand for about 10 minutes.

11.3.5 Transfer a suitable aliquot (11.3.4) from the solution to a suitable absorption cell and make the photometric readings using a light filter at 490 nm.

11.3.6 Calibration Curve

Take a series of standard iron solution and the reagent blank. Using the same quantities of reagents carry through all stages as described under 11.3.2 to 11.3.5 and plot the photometric readings of the standard solutions against milligrams of iron per 100 ml of the solution.

11.4 Calculation

Convert the photometric readings of the sample to mg of iron by means of calibration curve and calculate

the percentage of iron as follows:

$$11.4.1 \text{ Iron, percent by mass} = \frac{A \times 1}{B \times 10}$$

where

A = mass, in mg, of iron found in the aliquot; and

B = mass, in g, of the sample represented by the aliquot taken

$$11.4.2 \text{ Fe}_2\text{O}_3 \% = \text{Fe} \% \times 1.429$$

12 DETERMINATION OF SULPHATES BY THE GRAVIMETRIC METHOD

12.1 Outline of the Method

The sample is fused with sodium carbonate and the fused mass is treated with perchloric acid. The residue is dissolved in hydrochloric acid and sulphates precipitated as barium sulphate.

12.2 Reagents

12.2.1 Boric Acid, Solid.

12.2.2 Sodium Carbonate, Anhydrous.

12.2.3 Hydrochloric Acid, rd = 1.16 (Conforming to IS 265).

12.2.4 Ammonium Hydroxide, rd = 0.90.

12.2.5 Methyl Red Indicator

12.2.6 Barium Chloride Solution (10 Percent Aqueous Solution)

Dissolve 10 g of barium chloride in a little of water and dilute to 100 ml.

12.2.7 Dilute Sulphuric Acid, 1:1 (v/v).

12.3 Procedure

12.3.1 Weigh 2.10 g of sample in a platinum dish and fuse with 4 g of boric acid and 8 g of sodium carbonate, cool and add 50 ml of water and 25 ml of hydrochloric acid and heat to boiling. Cool and filter through fine textured filter paper into a 500 ml beaker. Wash twice with 50 ml water. Add 10 ml of water, neutralize excess acid with ammonia (add methyl red indicator for checking it to alkaline media). Add 5 ml hydrochloric acid excess and boil. Add 10 ml of barium chloride solution and boil it again, filter through medium textured filter paper and wash the precipitate with hot water till free from chloride. Transfer the filter paper with precipitate into pre-weighed platinum crucible with lid and incinerate at low temperature. Place the crucible and lid in a furnace and ignite the residue at $800 \pm 20^\circ \text{C}$. Allow the crucible to cool in a desiccator and

moist with 2 to 3 drops of dilute sulphuric acid. Heat slowly and ignite again at $800 \pm 20^\circ\text{C}$. Cool and weigh.

12.3.2 Blank

Carry out a blank determination following the procedure specified in 12.3.1 and using the same quantity of all the reagents but without the sample.

12.4 Calculations

Calculate the percentage of sulphate as follows:

$$\text{Sulphate (as SO}_3\text{), percent} = \frac{m_2 - m_1}{m} \times 41.145$$

where

m_1 = mass in g, of residue of barium sulphate, if any, obtained in the blank;

m_2 = mass in g, of barium sulphate residue obtained in the sample; and

m = mass in g, of sample taken.

13 DETERMINATION OF PHOSPHORUS PENTOXIDE AND SILICA BY SPECTROPHOTOMETRIC METHOD

13.1 Outline of the Method

Sample is fused with soda-boric acid mixture and fused mass is treated with water to make a definite volume. Suitable aliquots are taken for development of complex with ammonium molybdate at different pH. Phosphorus and silica are determined spectrophotometrically at 710 and 815 nm respectively.

13.2 Reagents

13.2.1 *Sodium Carbonate*, Anhydrous.

13.2.2 *Boric Acid*, Solid.

13.2.3 *Dilute Sulphuric Acid* 1:3 (v/v).

13.2.4 *Ammonium Molybdate*, 3 Percent (13.2.4.1) and 10 Percent (13.2.4.2).

13.2.4.1 Dissolve 3.0 g of ammonium molybdate in 60 ml of hydrochloric acid ($\text{rd} = 1.16$) and make up the volume to 100 ml. This solution is used for estimation of phosphorus.

13.2.4.2 Dissolve 10 g of ammonium molybdate in water and make up the volume to 100 ml in a volumetric flask. This solution is used for estimation of silica.

13.2.5 *Tartaric Acid*, 5 Percent (m/v).

13.2.6 *Ascorbic Acid*, 2 Percent (Prepare Immediately Before Use).

13.2.7 *Standard Phosphorus Solution* (1 ml = 0.01 mg P_2O_5)

Weigh 0.1917 g of dried potassium dihydrogen phosphate (KH_2PO_4), transfer to 1 litre volumetric flask and make up. Dilute 10 ml of the solution to 100 ml in a volumetric flask.

13.2.8 *Standard Silica Solution* (1 ml = 0.01 mg SiO_2)

Weigh 0.500 g of finely powdered pure quartz (previously ignited at 1000°C for 1 hour) in a platinum crucible. Add 5 g of sodium carbonate and mix. Fuse the mass at low temperature and finally at 750°C , till it forms in a clear melt. Cool, add little water and heat to dissolve the melt. Transfer the solution into polyethylene beaker, cool and dilute to 500 ml in a volumetric flask and make up. Transfer the solution immediately to polyethylene bottle. Transfer 10 ml of the solution to 1 litre volumetric flask and make up. Transfer the solution immediately to polyethylene bottle.

13.2.9 *Stannous Chloride Reducing Solution (for Phosphorus)*

13.2.9.1 *Stock solution*

Dissolve 10 g of stannous chloride in 25 ml of hydrochloric acid ($\text{rd} = 1.16$)

13.2.9.2 *Working solution*

Dilute 1.5 ml of stock solution (13.2.9.1) to 250 ml with distilled water.

13.3 Procedure

13.3.1 *Preparation of the Main Solution*

Weigh 1 g of sample, mix with 2 g of boric acid and 4 g of sodium carbonate in a platinum crucible. Incinerate the contents from the low bunsen flame to muffle furnace. Swirl the crucible and contents and heat again for 10 minutes. Remove the crucible from the furnace and cool. Fill the crucible with water. Heat gently until the entire melt has been dissolved and then cool the solution and transfer to the polyethylene beaker containing 50 ml water. Rinse the crucible with 10 to 20 drops of dilute hydrochloric acid and transfer to the beaker with few ml of water. Transfer the solution to 100 ml volumetric flask and make up. Transfer this solution immediately to a polyethylene bottle (Main Solution).

13.3.2 *For Phosphorus Pentoxide*

Pipette 50 ml aliquot of solution (13.3.1) into 100 ml beaker and adjust pH to 2.0 with dilute hydrochloric acid (1:3). Transfer to 100 ml volumetric flask and add 5 ml of hydroxylamine hydrochloride and 10 ml of ammonium molybdate solution 13.2.5.1. Swirl the solution. After 10 minutes, add 5 ml of stannous chloride reducing solution. Measure the absorbance of solution against reagent blank after 10 minutes at 710 nm.

13.3.2.1 Blank

Prepare a reagent blank by mixing 2 g of boric acid and 4 g of sodium carbonate. As described in preparation of sample solution (13.3.1), fuse the contents and make 100 ml. Take 50 ml aliquot and proceed in accordance with 13.3.2.

13.3.2.2 Calibration curve

Transfer 0, 2, 4, 6, 8 and 10 ml of standard phosphorus solution (13.2.7) into six 100 ml volumetric flasks. Proceed in accordance with 13.3.2 and plot the photometric readings of calibration solution, against mg of P_2O_5 per 100 ml solution. Convert the photometric readings of sample solution to mg of phosphorus pentoxide and calculate the phosphorus content as percent by mass of P_2O_5 in accordance with 13.3.4.

13.3.3 For Silica

Transfer 25 ml of the main solution to a 200 ml polyethylene beaker. Adjust the pH to 0.8-0.9. Add 10 ml of aqueous ammonium molybdate 13.2.4.2 and mix. Allow to stand for 15 minutes and mix. Add 5 ml of tartaric acid solution and 10 ml of dilute sulphuric acid (1 : 1) followed by 2 ml ascorbic acid. Swirl the solution after each addition. After 10 min transfer to 100 ml volumetric flask and make up. Measure the absorbance at 815 nm against reagent blank.

13.3.3.1 Blank

Take 25 ml aliquot of blank obtained under 13.3.2.1 and proceed in accordance with 13.3.3.

13.3.3.2 Calibration Curve

Transfer 0, 10, 20, 40, 50 and 60 ml of standard silica solution (13.2.8) to six 100-ml volumetric flasks, proceed in accordance with 13.3.3 and plot the photometric readings of solution against SiO_2 per 100 ml of solution. Convert the photometric readings of sample to mg of SiO_2 . Calculate the percentage of silica (as SiO_2) in accordance with 13.3.4.

13.3.4 Calculation

$$\text{Phosphorus (as } P_2O_5\text{),} \\ \text{percent by mass} = \frac{(A - B)}{C \times 5}$$

$$\text{Silica (as } SiO_2\text{),} \\ \text{percent by mass} = \frac{(A - B)}{C \times 2.5}$$

where

A = mass, in mg, of P_2O_5 or SiO_2 found in sample;

B = mass, in mg, P_2O_5 , or SiO_2 found in blank,;and

C = mass, in g, of sample taken for test.

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